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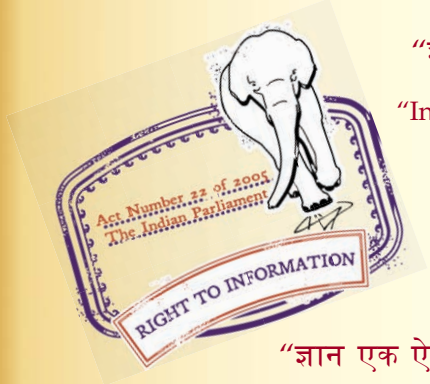
IS 8071 (1976): Propanil, Technical [FAD 1: Pesticides and Pesticides Residue Analysis]

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"Knowledge is such a treasure which cannot be stolen"

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IS : 8071 - 1976
( Reaffirmed 2012 )

# *Indian Standard*

# SPECIFICATION FOR PROPANIL, TECHNICAL

UDC 632·954 PRO



INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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# *Indian Standard*

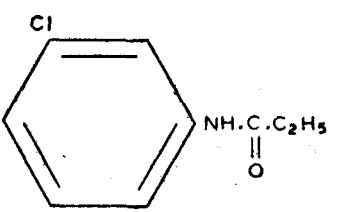
## SPECIFICATION FOR PROPANIL, TECHNICAL

## 0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 28 May 1976, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Propanil is a contact herbicide and recommended for the control of weeds.

0.3 Propanil is accepted common name by the International Organization for Standardization ( IS0 ) for N-( 3, 4-dichlorophenyl )-propionamide. The empirical and structural formulae and molecular mass are as given below:

| *Empirical Formula* | *Structural Formula* | *Mo tecutar Mass* |
|---|---|---|
| $C_9H_9NOCl_2$ |  | 218 |



*0.4* In the preparation of this standard due consideration has been given to the provisions of the Insecticides Act, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Rules for rounding off numerical values ( *revised* ).

## 1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for propanil, technical.

## 2. REQUIREMENTS

2.1 Description-The material shall essentially comprise N-( 3, 4-dichlorophenyl )-propionamide and shall be in the form of crystalline fused lumps, insoluble in water.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR PROPANIL, TECHNICAL

| Sl No. | Characteristic | Requirement | Method of Test, Rep to | |
|---|---|---|---|---|
| | | | Appendix of This Standard | Cl No. of IS : 6940-1973* |
| (1) | (2) | (3) | (4) | (5) |
| i) | Propanil content, percent by mass, *Min* | 89·0 | A | — |
| ii) | Chlorine content, percent by mass | 31·5 to 33-5 | B | — |
| iii) | Acidity ( as $H_2SO_4$ ), percent by mass, *Max* | 0·5 | — | 11.3.2 |

*Methods of tests for pesticides and their formulations.

## 3. PACKING AND MARKING

3.1 **Packing** — The material shall be packed in clean and dry mild steel containers.

3.2 Marking-The container shall be securely closed and shall bear legibly and indelibly the following in addition to the information as is necessary under the Insecticides Act and Rules:

a) Name of the material,
b) Name of the manufacturer,
c) Date of manufacture,
d) Batch number,
e) Propanil content,
f) Net mass of the contents, and
g) The minimum cautionary notice worded as under:

'HAZARDOUS. KEEP AWAY FROM FOODSTUFFS AND ANIMAL FEED. DO NOT USE CONTAINERS FOR STORAGE OF FOODSTUFF AND ANIMAL FEED. WASH HANDS THOROUGHLY WITH SOAP AFTER HANDLING.'

**3.2.1 The containers may also be marked with the ISI Certification Mark.**

NOTE -The use of the IS1 Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The IS1 Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by IS1 and operated by the producer. IS1 marked products are also continuously checked by IS1 for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the IS1 Certification Mark may be granted to manufacturers or processors, may he obtained from the Indian Standards Institution.

## 4. SAMPLING

**4.1 Representative samples shall be drawn as prescribed in 'Indian Standard methods for sampling of pesticides and their formulations (*under preparation*)'.**

NOTE — Till such time the standard under preparation is published, the matter shall be as agreed to between the parties concerned.

## 5. TESTS

**5.1 Tests shall be carried out as prescribed under col 4 and 5 of Table 1.**

**5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (see IS :1070-1960* ) shall be employed in tests.**

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

# APPENDIX A
## [ Table 1, *Item* (i) ]

### DETERMINATION OF PROPANIL CONTENT

### A-O. GENERAL

**A-O.1 Either of two methods, namely, gas chromatographic method and** titration **method, may be used for determination of propanil content.**

### A-l. GAS CHROMATOGRAPHIC METHOD

**A-I.1 Summary of the Method-The method consists of injecting a sample along with an internal standard in a known proportion into a gas** chromatograph **and determining the area under each peak. The area under the peak is proportional to the mass of sample. By comparison of this area with that of the standard the percentage purity of the sample is determined.**

*Specification for water, distilled quality ( revised).

### A-1.2 Apparatus

**A-1.2.1** *Gas-Liquid Chromatograph* — equipped with a recorder and disc integrator and fulfilling the following conditions:

| | |
|---|---|
| Column | 1·828 m long |
| Detector | Thermal conductivity type |
| Column temperature | 230°C, isothermal |
| Injection port temperature | 280 ± 5°C |
| Detector temperature | 300 ± 5°C |
| Flow rate | 30 ml/min |
| Chart speed | 760 mm/h |

A-1.2.2 *Column* — consisting of 1'828 m stainless steel (designation 04Cr14Ni10) tubing, of 3·175 mm outer diameter and 2'362 mm internal diameter, packed with 3'6 g of 10 percent GE-SE-30 on 150-180 Micro Gas Chrom Q.

A-1.2.3 *Micro-syringe* — 10 µl capacity.

### A-1.3 Reagents

**A-1.3.1** *Nitrobenzene* — pure by gas chromatography.

A-1.3.2 *2, 4-Dichlorophenyl-4'-Nitrophenyl Ether, Purified* — Add 100 g of molten 2, 4-dichlorophenyl-4'-nitrophenyl ether to 850 ml ethyl alcohol warmed to 70°C. Stir the contents to dissolve and filter the hot solution through filter paper. Cool the filtrate in an ice-bath for two hours and filter the crystals under suction. Dissolve these crystals in 700 ml of ethyl alcohol and recrystallize a second time. Allow crystals to drain. thoroughly. Dry the crystals in a vacuum oven at 45°C until free from alcohol. The resultant 2, 4-dichlorophenyl-4'-nitrophenyl ether should be 100 percent pure by GLC.

A-1.3.3 *3,4-Dichloropropanilide, Purified* — Dissolve 40 g of 3,4-dichloropropanilide in about 400 ml benzene, add 50 ± 0'1 g 'Florisil' to the solution and filter under suction. Add petroleum ether to the filtrate until the solution is just turbid. About 400 to 600 ml of petroleum ether will be required. Place the beaker in a refrigerator at approximately 4°C for two hours. Filter the crystals under suction and allow to drain thoroughly. Dry the crystals under vacuum at room temperature until free of benzene. The purified crystals should be of not less than 99 percent purity.

A-1.3.4 *Internal Standard Solution-Weigh* accurately 5 ± 0·001 g of purified 2, 4-dichlorophenyl-4'-nitrophenyl ether (A-1.3.2) in a 150-ml beaker, and add accurately weighed 20·0 ± 0'001 g nitrobenzene into the same beaker. Stir, to dissolve the crystals and store in a glass stopper bottle.

## A-l.4 Procedure

**A-1.4.1** *Calibration-Weigh* accurately 0'200 ± 0'001 g of purified **3,4-dichloropropanilide** ( A-1.3.3 ) into a weighing bottle, and add accurately weighed 0'800 ± 0·001 g nitrobenzene into the same bottle. Shake the bottle to dissolve the crystals. Weigh out accurately about 0·5 g of this solution into another weighing bottle and add accurately about 0·5 g of internal standard solution and mix thoroughly. Inject about 3µl or any suitable quantity into the gas chromatograph so that all the peaks except that of nitrobenzene are within scale. Determine the number of disc integrator trace counts under each peak.

A-1.4.2 ***Analysis of Sample-Weigh*** accurately 1.000 ± 0'001 g of molten sample into a weighing bottle. Weigh into the same bottle 4'000 ± 0'001 g of nitrobenzene. Shake the contents till the sample dissolves completely. Warm if necessary. Weigh out accurately about 1'0 g of the sample solution into another weighing bottle, and add accurately about 1·0 g of the internal standard solution and mix thoroughly. Inject a suitable quantity of the sample into the gas chromatograph in order to get all the peaks except nitrobenzene within scale. Determine the number of disc integrator trace counts under each peak.

## A-l.5 Calculation

**A-1.5.1** Propanil content, percent by mass $= \frac{100\ M_1\ a}{Mfa_1}$

where

$M_1$ = mass in grams of the internal standard solution taken along with the sample,

$a$ = number of disc integrator trace counts under the sample peak,

$M$ = mass in grams of the sample solution taken for analysis,

$f = \frac{a_2 m_2}{a_2 m_1}$,

$a_1$ = number of disc integrator trace counts under the peak of the internal standard added to the sample,

as = number of disc integrator trace counts under the peak of the standard sample,

$m_2$ = mass in grams of the internal standard solution taken along with the standard sample solution,

$a_2$ = number of disc integrator trace counts under the peak of the internal standard added to the standard sample, and

$m_1$ = mass in grams of the standard sample solution taken for calibration.

## A-2. TITRATION METHOD

**A-2.1 Principle-** Propanil on hydrolysis yields 3, 4-dichloroaniline, which is estimated by titration against sodium nitrite.

A-2.2 **Reagents**

**A-2.2.1** *Standard Sodium Hydroxide Solution in Ethylene Glycol* — 1 N.

A-2.2.2 *Hydrochloric Acid* — 1 : 1 (see IS : 265-1962*).

*A-2.2.3 Standard Sodium Nitrite Solution* — 0·1 *N*.

*A-2.2.4 Phenolphthalein Indicator Solution*- 1 percent in neutral rectified spirit.

A-2.3 **Procedure-Weigh** accurately a sample containing 0·45 to 0·55 g of active ingredient in a flask. Add 25 ml of standard sodium hydroxide solution and reflux on a hot plate for one hour. Cool and wash the condenser with 10 to 15 ml distilled water and transfer the contents quantitatively into a 400-ml beaker. Add one or two drops of phenolphthalein indicator solution and neutralize with hydrochloric acid. Add 15 ml concentrated hydrochloric acid. Cool to about 0-5°C by adding ice and add 5 g of potassium bromide. Then titrate with standard sodium nitrite solution using potassium iodide starch indicator paper.

A-2.3.1 Carry out a blank determination.

*A-2.4* **Calculation**

**A-2.4.1** Propanil content, percent by mass $= \frac{10{\cdot}9\ (V - v)\ \mathcal{N}}{M}$

where

$V$ = volume in ml of standard sodium nitrite solution consumed for the test,

$v$ = volume in ml of standard sodium nitrite solution consumed for the blank determination,

$\mathcal{N}$ = normality of standard sodium nitrite solution, and

$M$ = mass in g of the material taken for the test.

# APPENDIX B

## [*Table* 1, *Item* (ii)]

## DETERMINATION OF TOTAL CHLORINE

### B-1. APPARATUS

**B-1.1 Parr-Peroxide Bomb and Ignition Assembly**

**B-1.2 Syringe** — l-ml injection syringe with needle.

*Specification for hydrochloric acid ( *revised* ).

## B-2. REAGENTS

**B-2.1 Concentrated Nitric Acid--See IS: 264-1968*.**

**B-2.2 Ferric Alum Indicator** -Dissolve 350 g of ferric ammonium sulphate crystals in 700 ml water, heating gently if necessary. Add 50 ml concentrated nitric acid, dilute to one litre with water and mix thoroughly.

B-2.3 Nitrobenzene — technical grade.

B-2.4 **Standard Silver Nitrate Solution** — 0·1 N.

B-2.5 Standard Ammonium Thiocyanate Solution — 0·1 N.

B-2.6 Thymol Blue Indicator-Dissolve 1·0 g of thymol blue powder in 500 ml alcohol and then dilute the solution with 500 ml water.

B-2.7 **Sodium** Peroxide-reagent grade. The material shall be of such fineness that it passes through 355-micron IS Sieve ( see IS: 460-1962† ).

B-2.8 Potassium Nitrate — Sugar Mixture- Dry 900 g of potassium nitrate, reagent grade, in an oven maintained at 120°C for 48 hours and pulverize the dry crystals. Simultaneously, dry 300 g of granulated sugar in an oven at 60°C for 48 hours, then pulverize the dry sugar. Thoroughly mix the pulverized potassium nitrate and sugar in a ball mill for 30 minutes.

## B-3. PROCEDURE

B-3.1 Add 1 g of potassium nitrate — sugar mixture into a clean dry fusion cup of the Parr-Peroxide bomb. Add approximately 15 g of sodium peroxide to the fusion cup and thoroughly mix the contents with a clean glass rod. Fill a clean dry I-ml syringe to the mark with the sample and withdraw the sample from the tip by pulling the plunger of the syringe slightly out. Wipe any material on the outside of the syringe with a piece of tissue paper and weigh the filled syringe to 0·1 mg. Insert the needle in the fusion mixture to a depth of 1·5 cm and slowly inject a volume of sample equivalent to 0·2-0·25 g. Wipe the needle with a 1'5 x 3·0 cm piece of filter paper and add the paper to the contents of the fusion cup and cover the cup with the bomb cap, then reweigh the syringe to 0·0001 g. Place the fusion cup in the bomb casing, assemble the bomb and tighten the lock nut. Shake the bomb vigorously for one minute. Pour l-2 ml of water on the bomb cap and set the bomb in the opening of the ignition housing. Adjust the burner to produce a narrow hot flame impinging on the bottom of

*Specification for nitric acid ( *first* revision ).
†Specification for test sieves ( *revised* ).

the bomb. Heat the bomb continuously care being taken not to expose the face or arms directly over the bomb. Continue heating till the contents are ignited as indicated by the boiiing of the water on the cap or until it heats for 8 minutes. Immediately after ignition, cool the bomb by placing it in a water bath. When cooled, remove from the water bath and rinse the outside of the bomb with distilled water. Remove the cap of the bomb and place the fusion cup on its side in a 400-ml beaker. Wash the cap with hot distilled water using enough water that will cover the fusion cup to half its diameter, then cover the beaker with a watch glass. When the effervescence has ceased, carefully remove the fusion cup with clean tongs and rinse with small amount of hot distilled water collecting the rinsings in the beaker. This solution should be colourless. In case the solution exhibits presence of yellow or brown colour, which indicates incomplete ignition which will interfere in the accuracy of the analysis, discontinue the determination and repeat the experiment starting with a fresh sample. Cool the contents of the beaker and add 1: 1 nitric acid with stirring until the effervescence becomes less vigorous as the acid is added. Then add ten drops of thymol blue indicator and sufficient 1: 1 nitric acid to turn the indicator yellow or very faint pink. Add an additional 10 ml of 1 : 1 nitric acid and 50 ml of standard silver nitrate solution. Thoroughly mix the contents of the beaker, add 2 ml of ferric alum and 10 ml of nitrobenzene. Stir vigorously and titrate the excess of silver nitrate with standard ammonium thiocyanate solution with vigorous stirring of the solution in the beaker until the appearance of a faint but permanent pink end point.

**B-3.1.1** Carry out a blank determination without the sample.

## B-4. CALCULATIONS

B-4.1 Total chlorine, percent by mass = $\frac{(A - B)\ 3'546}{M}$

where

A = volume in ml of silver nitrate used for sample x 0·1 N — volume of ammonium thiocyanate solution used for sample x 0·1 N.

$B$ = volume in ml of silver nitrate used for blank x 0·1N — volume of ammonium thiocyanate solution used for blank X 0·1 N.

B-4.2 Propanil content, percent by mass = $\frac{T \times 94{\cdot}5}{32{\cdot}7}$

where

$T$ = percent by mass of total chlorine in the sample.

( *Continued from page 2* )

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## PESTICIDES ( TECHNICAL GRADE )

IS:

1486-1969 Copper oxychloride, technical ( *first revision* )
1488-1969 2, 4-D sodium, technical ( *first revision* )
1682-1973 Cuprous oxide, technical ( fungicidal grade ) ( *first revision* )
1832-1961 Malathion, technical ( *first revision* )
1833-1961 Diazinon, technical
2125-1962 Phenyl mercury salicylate, technical
2126-1973 Phenyl mercury acetate, technical ( *first revision* )
2353-1963 Phenyl mercury chloride, technical
2354-1963 Ethyl mercury chloride, technical
2863-1964 Chlordane, technical
3898-1966 Zineb, technical
3900-1975 Ziram, technical ( *first revision* )
3902-1975 Dimethoate, technical ( *first revision* )
4320-1967 Thiram, technical
4321-1967 2, 4-D technical
4344-1967 Endosulfan, technical
4345-1967 Binapacryl, technical
4451-1967 Toxaphene, technical
4929-1968 Dichlorvos, technical
4958-1968 Phosphamidon, technical
5278-1969 Dicofol, technical
5280-1969 Fenitrothion, technical
6432-1972 Heptachlor, technical
7158-1973 Isobornyl thiocyanoacetate ( thanite ), technical
7539-1975 Carbaryl, technical
7945-1976 Trichlorfon, technical
7949-1976 Quintozene, technical
7950-1976 Fenthion, technical
7976-1976 Phorate, technical
7977-1976 Disulfoton, technical
8072-1976 Qunalphos, technical

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